3-Phenyl-4H-furo[3,2-c]chromen-4-one

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The crystal structure of the title compound, $C_{17}H_{10}O_3$, is the first example of a furocoumarin containing three fused rings. The tricyclic furocoumarin fragment is perfectly planar. The phenyl substituent forms a dihedral angle of 39.52 (8)° with the plane of the tricyclic system. The crystal packing involves centrosymmetric dimers interconnected by strong π-interactions between their furo[3,2-c]coumarin fragments [at distances of 3.42 (4) Å].

Comment

Linear or angular furocoumarins are natural products showing a wide range of biological properties (Zubia *et al.*, 1992). This class of compounds has been of synthetic interest over the years (Singh & Singh, 1985; Majumdar *et al.*, 1988; Laschober & Kappe, 1990; Saidi & Bigdeli, 1998, and references therein). In the course of ongoing studies of chromenone-containing heterocyclic systems of biological importance, we have synthesized furo[3,2-c]coumarin derivatives from 4-hydroxy-coumarin and α-haloketones *via* O-alkylation and subsequent cyclization *in situ* (Risitano *et al.*, 2001). We describe here a novel crystal structure of a representative example of these interesting compounds, namely 3-phenylfuro[3,2-c]chromen-4-one, (I). A perspective view of (I) with the atom-numbering scheme is shown in Fig. 1.

(I)
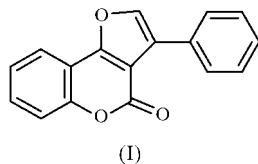
The furo[3,2-c]coumarin fragment and the phenyl ring bonded to C10 are both planar within experimental error. The phenyl ring is rotated by 39.52 (8)° with respect to the furo-coumarin fragment and the C10—C12 bond distance of 1.482 (3) Å denotes the absence of conjugation between the two π-delocalized systems. This orientation is stabilized by a weak intramolecular hydrogen bond linking the carbonyl O and H17 atoms [O2···C17 = 3.197 (3) Å and C17—H17···O2 = 117°].

The bond distances and angles of the coumarin moiety are in good agreement with the corresponding values reported for such fragments in the Cambridge Structural Database (CSD; Allen *et al.*, 1991). The endocyclic furan ring bond distances indicate π-electron delocalization existing over the whole tricyclic system.
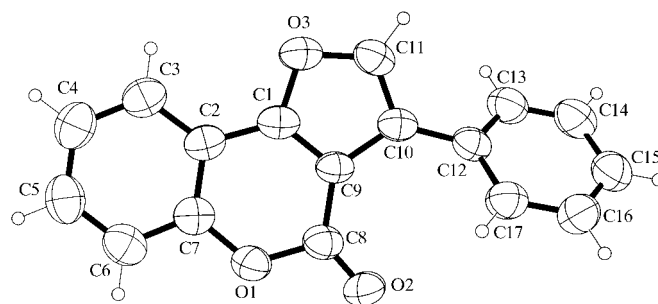

Figure 1
Perspective view of the title compound showing the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

Here, as well as in other coumarin compounds reported in the April 2000 (Version 5.19) release of the CSD, an important asymmetry in the O—C—O bond angles has been detected [O2—C8—O1 = 115.6 (2)° and O2—C8—C9 = 129.6 (3)°]. Even more significant differences have been observed in other molecules containing the furan-5-one fragment, as we have already reported (Risitano *et al.*, 1997). In order to rationalize the electronic and steric factors that determine this asymmetry, we have begun statistical studies and *ab initio* calculations on suitable model fragments (Bruno & Nicoló, unpublished results).
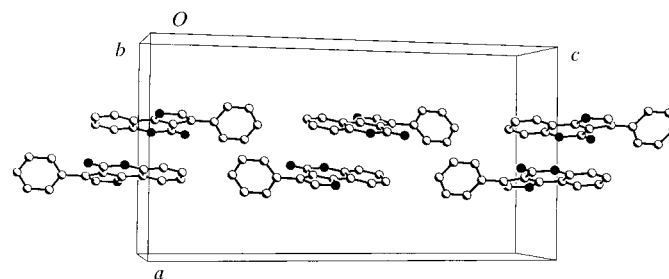

Figure 2
The crystal packing of the title compound showing the π-stacking between the centrosymmetric molecules. Atom size is arbitrary.

In the crystal lattice, each pair of molecules related by an inversion centre shows a π-interaction of 3.42 (4) Å between their planar furo[3,2-c]coumarin fragments (average distance of the atoms of one fragment from the plane of the other fragment). Despite the out-of-plane rotation, the phenyl rings are situated on the external side of the overlapping centro-symmetric dimers and do not hinder their strong π-stacking. Several weak hydrogen bonds involving the O atoms are also responsible for the molecular packing.

organic compounds

Experimental

The title compound was obtained as described previously (Risitano *et al.*, 2001) and was recrystallized from methanol.

Crystal data

$C_{17}H_{10}O_3$
$M_r = 262.25$
Orthorhombic, $Pbca$
$a = 13.4239 (11)$ Å
$b = 7.2198 (5)$ Å
$c = 25.441 (3)$ Å
$V = 2465.7 (4)$ Å^3
$Z = 8$
$D_x = 1.413$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 48
  reflections
$\theta = 3.04–12.36°$
$\mu = 0.097$ mm^{-1}
$T = 298 (2)$ K
Irregular, colourless
$0.39 \times 0.25 \times 0.18$ mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: ψ scan
  (Kopfmann & Huber, 1968)
  $T_{min} = 0.241$, $T_{max} = 0.272$
2877 measured reflections
2170 independent reflections
1189 reflections with $I > 2\sigma(I)$

$R_{int} = 0.029$
$\theta_{max} = 25.0°$
$h = -1 \rightarrow 15$
$k = -1 \rightarrow 8$
$l = -30 \rightarrow 1$
3 standard reflections
  every 197 reflections
  intensity decay: 12%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.123$
$S = 0.980$
2170 reflections
182 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0530P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Extinction correction: $SHELXL$97
Extinction coefficient: 0.0057 (7)

H atoms were placed in calculated positions (their idealized geometry depended on the parent C-atom type) and were included in the refinement as riding atoms with a common fixed isotropic displacement parameter ($U_{iso} = 0.075$ Å^2).

Data collection: *XSCANS* (Siemens, 1996a); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1996b); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XPW* in *SHELXTL*; software used to prepare material for publication: locally modified *PARST*97 (Nardelli, 1995) and *SHELXL*97.

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.385 (3)	O3—C11	1.383 (3)
O1—C8	1.403 (3)	C1—C9	1.357 (3)
O2—C8	1.196 (3)	C10—C12	1.482 (3)
O3—C1	1.358 (3)		
C7—O1—C8	123.8 (2)	O2—C8—O1	115.6 (2)
C1—O3—C11	105.14 (19)	O2—C8—C9	129.6 (3)
C9—C1—O3	111.0 (2)	O1—C8—C9	114.8 (2)
O3—C1—C2	123.6 (2)		
C9—C10—C12—C17	−43.2 (4)		

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV1062). Services for accessing these data are described at the back of the journal.

References

Allen, F. H., Davies, J. E., Galloy, J. J., Johnson, O., Kennard, O., Macrae, C. F., Mitchell, E. M., Mitchell, G. F., Smith, J. M. & Watson, D. G. (1991). *J. Chem. Inf. Comput. Sci.* **31**, 187–204.
Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Kopfmann, G. & Huber, R. (1968). *Acta Cryst.* **A24**, 348–351.
Laschober, R. & Kappe, T. (1990). *Synthesis*, pp. 387–388.
Majumdar, K. C., De, R. N., Khan, A. T., Chattopdhyay, S. K., Dey, K. & Patra, A. (1988). *J. Chem. Soc. Chem. Commun.* pp. 777–779.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Risitano, F., Grassi, G., Bruno, G. & Nicoló, F. (1997). *Liebigs Ann. Chem.* pp. 441–445.
Risitano, F., Grassi, G., Foti, F. & Bilardo, C. (2001). *Tetrahedron Lett.* Submitted.
Saidi, M. R. & Bigdeli, K. (1998). *J. Chem. Res. (S)*, pp. 800–801.
Sheldrick, G. M. (1997). *SHELXL*97. Release 97–2. University of Göttingen, Germany.
Siemens (1996a). *XSCANS*. Release 2.20. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996b). *SHELXTL*. VMS Release 5.05. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Singh, R. P. & Singh, D. (1985). *Heterocycles*, **23**, 903–907.
Zubia, E., Luis, F. R., Massanet, G. M. & Collado, I. (1992). *Tetrahedron*, pp. 4239–4246.